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Microstructural and mechanical characterization of contemporary lingual orthodontic brackets

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Abstract: Objectives: To investigate the composition and the microstructural and mechanical characterization of three different types of lingual brackets. Materials and Methods: Incognito™ (3M Unitek), In-Ovation L (DENTSPLY GAC) and STb™ (Light Lingual System, ORMCO) lingual brackets were studied under the scanning electron microscope employing backscattered electron imaging and their elemental composition was analysed by energy-dispersive X-ray microanalysis. Additionally, Vickers hardness was assessed using a universal hardness-testing machine, and the indentation modulus was measured according to instrumented indentation test. Two-way analysis of variance was conducted employing bracket type and location (base and wing) as discriminating variable. Significant differences among groups were allocated by post hoc Student-Newman-Keuls multiple comparison analysis at 95% level of significance. Results: Three different phases were identified for Incognito and In-Ovation L bracket based on mean atomic number contrast. On the contrary, STb did not show mean atomic contrast areas and thus it is recognized as a single phase. Incognito is a one-piece bracket with the same structure in wing and base regions. Incognito consists mainly of noble metals while In-Ovation L and STb show similar formulations of ferrous alloys in wing and base regions. No significant differences were found between ferrous brackets in hardness and modulus values, but there were significant differences between wing and base regions. Incognito illustrated intermediate values with significant differences from base and wing values of ferrous brackets. Conclusions/Implications: Significant differences exist in microstructure, elemental composition, and mechanical properties among the brackets tested; these might have a series of clinical implications during mechanotherapy.

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Microstructural and mechanical characterization of contemporary lingual orthodontic brackets

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SUMMARY

OBJECTIVES: To investigate the composition and the microstructural and mechanical characterization of three different types of lingual brackets.

MATERIALS AND METHODS: Incognito™ (3M Unitek), In-Ovation L (DENTSPLY GAC) and STb™ (Light Lingual System, ORMCO) lingual brackets were studied under the scanning electron microscope employing back-scattered electron imaging and their elemental composition was analysed by energy-dispersive X-ray microanalysis. Additionally, Vickers hardness was assessed using a universal hardness-testing machine, and the indentation modulus was measured according to instrumented indentation test. Two-way analysis of variance was conducted employing bracket type and location (base and wing) as discriminating variable. Significant differences among groups were allocated by *post hoc* Student-Newman-Keuls multiple comparison analysis at 95% level of significance.

RESULTS: Three different phases were identified for Incognito and In-Ovation L bracket based on mean atomic number contrast. On the contrary, STb did not show mean atomic contrast areas and thus it is recognized as a single phase. Incognito is a one-piece bracket with the same structure in wing and base regions. Incognito consists mainly of noble metals while In-Ovation L and STb show similar formulations of ferrous alloys in wing and base regions. No significant differences were found between ferrous brackets in hardness and modulus values, but there were significant differences between wing and base regions. Incognito illustrated intermediate values with significant differences from base and wing values of ferrous brackets.

CONCLUSIONS/IMPLICATIONS: Significant differences exist in microstructure, elemental composition, and mechanical properties among the brackets tested; these might have a series of clinical implications during mechanotherapy.

Introduction

Lingual bracket systems nowadays are available in a variety of different treatment options: conventional or self-ligating systems, made from stainless steel, nickel-free Co–Cr or Au alloys, pre-fabricated or fully customized with horizontal or vertical slots. Most of them have a 0.018 inch slot, since the increased stiffness of heavier archwires is undesirable in the anterior region of lingual appliances, where the interbracket distance is decreased.

Elemental composition of these alloys influences their biocompatibility, corrosion resistance, and ionic release (Knoernschild *et al.*, 1999; Staffolani *et al.*, 1999; Locci *et al.*, 2000; Karov & Hinberg, 2001; Huang *et al.*, 2004) as well as their biomechanical performance. Co–Cr alloys increase the frictional coefficients, in comparison with stainless steel (Kusy *et al.*, 1991). In contrast with this, surface and frictional analyses of aesthetic brackets with 18 kt Au-lined (Kusy and Whitley, 2001) or Au–Pd slot (Doshi and Bhad-Patil, 2011) render them a good alternative to

stainless steel in space closure with sliding mechanics. Moreover, increased slot hardness is essential in order to avoid binding of the wire onto the bracket slot walls, which could increase friction, and as a result, higher force magnitudes are necessary in order to accomplish tooth movement (Eliades, 2011).

There is no information regarding the structural and mechanical characteristics of lingual brackets, and unfortunately, their clinical performance cannot be inferred from the existing literature data about labial appliances, due to biomechanical (Geron *et al.*, 2004), structural (Wiechmann, 2002), or technique inherent differences (Demling *et al.*, 2009). Therefore the aim of this study was to investigate the composition, the microstructure, and mechanical properties of three different types of lingual brackets.

Materials and methods

The following three bracket types were evaluated: Incognito™ lingual brackets (3M Unitek, Monrovia,

California, USA, Lot 106332), In-Ovation L lingual brackets (DENTSPLY GAC, Bohemia, New York, USA, Lot S000512000) and STb™ lingual brackets (Light Lingual System, ORMCO, Orange, California, USA, Lot HS Code 9021.10.0090). These brackets were previously evaluated *in vitro* regarding torque delivery, with a procedure, which included the ligation of 10 rectangular archwires (Sifakakis *et al.*, 2013).

Five upper premolar brackets from each bracket group (15 brackets in total) were embedded in epoxy resin (Epofix, Struers Copenhagen Denmark), ground with water coolant SiC papers from 220 to 2000 grit, and polished up to 1 µm alumina slurry (Buehler, Lake Bluff, Ill) in a grinding/polishing machine (Ecomet III, Buehler). Then the specimens were ultrasonically cleaned for 5 minutes and vacuum coated with a thin layer of conductive carbon. The surface of cross sections were studied under the scanning electron microscope employing backscattered electron imaging (BEI), and their elemental composition was analysed by energy-dispersive X-ray microanalysis employing a Si(Li) energy-dispersive spectroscopic (EDS) detector (Sapphire, EDAX, Mahwah, New Jersey, USA) with super ultrathin window (Be). The X-ray EDS (EDX) spectra were acquired from the wing and base regions of the cross-section under 30kV accelerating voltage and 98 µA beam current using an area analysis mode at 1000× magnification, a 130 × 130 µm sampling window, and 200 second acquisition time. The quantitative analysis was performed by Genesis software (version 5.1, EDAX) under a non-standard analysis, using ZAF (atomic number–absorption–fluorescence) correction methods.

The same specimens were repolished, and the surfaces were used for the assessment of Vickers hardness ($HV_{0.05}$), using universal hardness-testing machine ZHU0.2/Z2.5 (Zwick Roell, Ulm, Germany) applying a load of 5 N and 12 second contact time. One reading was taken from each specimen. In addition, indentation modulus (E_{IT}) was measured according to standardized test method (ISO 14577-1, 2002). This technique requires the simultaneously monitoring of load and indentation depth during loading–unloading cycle, and E_{IT} estimation is based on the initial slope from unloading data. The determination was based according to mathematical formulas provided by ISO 14577-1 employing the following formula:

$$E_{IT} = \frac{1 - (v_s)^2}{\frac{1}{E_r} - \frac{1 - (v_i)^2}{E_i}} \quad (1)$$

Where, v_s (0.3) and v_i (0.07) the Poisson's ratio of test piece and indenter, respectively; E_i the modulus of the indenter (1140 GPa); E_r the reduced modulus given by the formula:

$$E_r = \frac{\sqrt{\pi}}{2C\sqrt{A_p}} \quad (2)$$

Where, C denotes the compliance of the contact and is determined by the slope of dh/dF at maximum test force; and A_p is the projected contact area defined in accordance with ISO 14577-2. (ISO 14577-2, 2002).

The results of hardness and modulus tests were statistically analysed by two-way analysis of variance employing bracket type and location (base and wing) as discriminating variables. Significant differences among groups were allocated by *post hoc* Student-Newman-Keuls multiple comparison analysis at 95% level of significance.

Results

Figure 1 shows representative BEI from the wing region of brackets included in the study. Figure 1a demonstrates the Incognito structure with intermediate mean atomic number contrast matrix (M) and a lighter randomly dispersed almost circular phase (G). Interestingly a heavier phase was identified at the vicinity or within the volume of this dispersed phase (W). Three different phases were also identified for the base region of In-Ovation L bracket based on mean atomic number contrast in Figure 1b. A diffuse distribution of a lighter phase (G) in the matrix (M). Almost circular tiny phases (W) were identified at the interface between (M) and (G) as shown in the inset picture of Figure 1b. Contrarily, STb did not show mean atomic contrast areas and thus it is recognized as a single phase. The latter is true for the wing region of STb and In-Ovation L. Incognito is a one-piece bracket with the same structure in wing and base regions.

The elemental content for all brackets tested after EDX analysis is shown in Table 1. For the purpose of clarity, only the mean values of each element are presented. Incognito consists mainly of noble metals while In-Ovation L and STb show similar formulations of ferrous alloys in wing and base regions.

Figure 2 exhibits representative hardness indentations in wing and base regions with softer alloys having a larger indentation mark as readily shown in this image. Representative force versus indentation depth curve for all materials tested is presented in Figure 3 with dot line showing the tangent to unloading curve at maximum force, while Figure 4 depicts the results of hardness and E_{IT} for all brackets tested. As Incognito is a one-piece bracket the same values of hardness and E_{IT} was appended to base and wing regions. No significant differences were found between ferrous brackets in hardness and modulus values, but there were significant differences between wing and base regions. Incognito illustrated intermediate values with significant differences from base and wing values of ferrous brackets.

Discussion

The two ferrous brackets (In-Ovation L and STb) depicted only microstructural differences between each other with

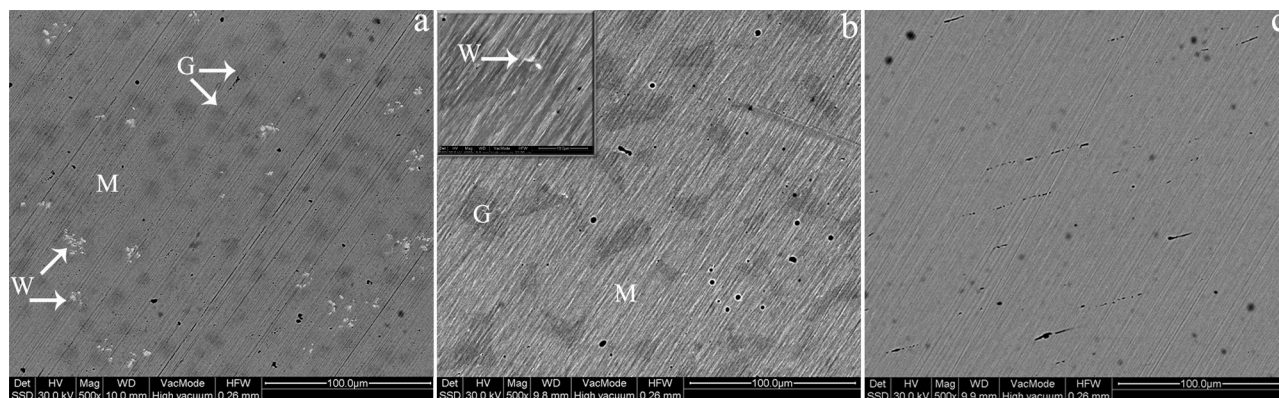


Figure 1 Representative backscattered electron images from the cross-section of bracket tested. (a) Incognito: a diffuse distribution in a matrix (M) of almost circular lower mean atomic number phase (G) with few of them including a higher mean atomic number phase (W). (b) In-Ovation L with a diffuse distribution of almost polygon regions of lower mean atomic number. Inset figure demonstrates, in higher magnification, the development of a heavier phase (W) at the interface between matrix (M) and diffuse phase (c) STb: a single phase without mean atomic differences. Gray circular spots are attributed to surface contamination as shown in respective secondary electron image (not shown here) [Original magnification $\times 500$ (inset $\times 4000$). Bar $100\ \mu\text{m}$ (inset $10\ \mu\text{m}$)].

Table 1 Quantitative results after X-ray energy-dispersive spectroscopic analysis. Mean values from three measurements. Standard deviations (SD) are less than 0.4 for all elements tested. SDs are not shown for the sake of clarity. W, wing; B, base.

Brackets	Location	Fe	Cr	Ni	Cu	Mo	Mn	Si	Al	Au	Ag	Pt	Zn
Incognito	W/B				21.3			0.5		57.9	13.1	6.5	0.5
In-Ovation L	W	72.1	17.7	4.1	4.1		0.8	1.0	0.2				
	B	65.4	18.4	11.2	0.3	2.3	0.9	0.6	0.2				
STb	W	74.3	16.4	4.1	3.9		1.0	0.4					
	B	66.3	17.8	10.9	0.3	2.2	1.5	0.7	0.2				

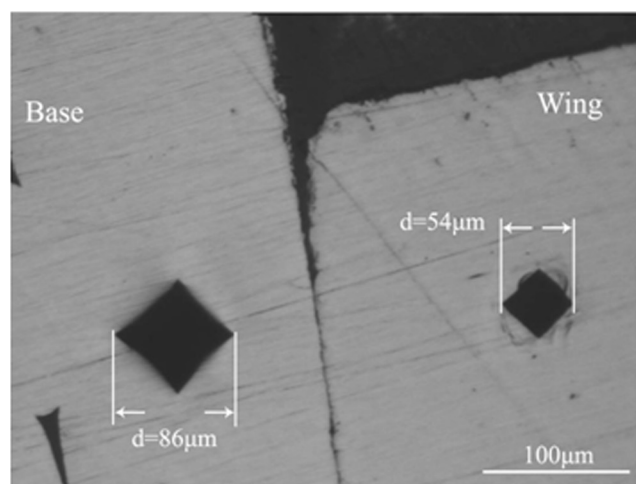


Figure 2 Cross-section image illustrating indentation impression on softer base and harder wing alloys. The interface between the two parts is also readily shown in the middle (Bar $100\ \mu\text{m}$).

Incognito showing substantial differences with the ferrous brackets in microstructure, elemental composition, and mechanical properties. As two-piece structure, In-Ovation L and STb brackets consist of different alloys in wing and base parts. For both brackets the elemental composition

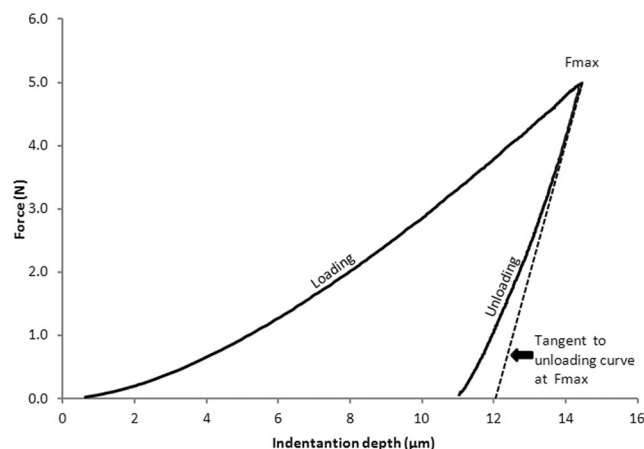


Figure 3 Representative curve of a force-indentation depth of brackets tested with loading-unloading cycle. Tangent to unloading curve at maximum force (F_{max}) is also shown with dotted line.

of alloys used for the base corresponds to the nominal composition (%wt: Fe: balance, Mn: 2.0, Cr: 16–18, Ni: 10–14, Mo: 2–3, and traces of P, S, and C) of an austenitic stainless steel AISI 316 alloy (Darabara *et al.*, 2007) while the alloy for the wing fits within the limits of martensitic precipitation hardening stainless steel alloy with nominal

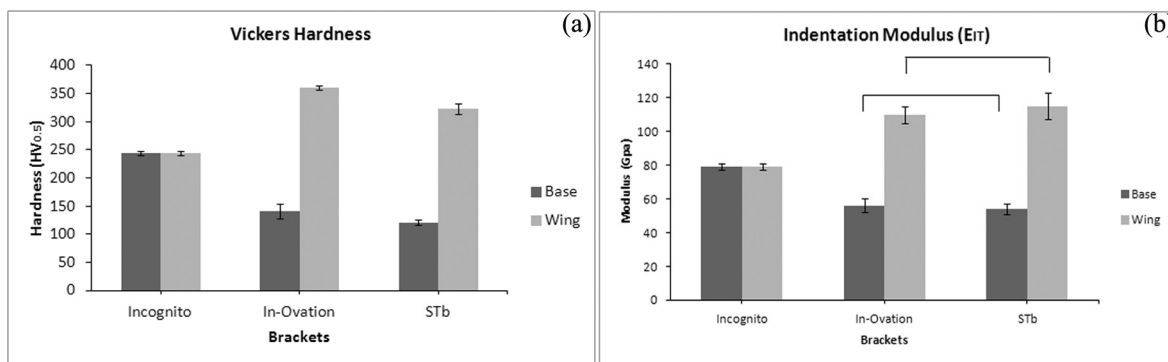


Figure 4 Graphs showing the Vickers hardness (a) and indentation modulus (b) for all brackets tested. The same mean values and standard deviations were appended to Incognito for base and wing regions as it is a one-piece structure. Connecting lines imply mean values without statistical significant differences ($P > 0.05$).

composition of %wt: Fe: balance, Cr: 15–17.5Cr, Ni: 3–5, Cu: 3–5, Si:1, Mn:1 and traces of P, S, and C (Siargos *et al.*, 2007). The alloys used for the production of base did not show mean atomic contrast and thus, are characterized as single phase alloys. On the contrary, and despite the similarity in their elemental compositions, the wing alloys illustrated completely different microstructures—a finding that is attributed probably to variations in thermal treatment to achieve precipitation hardening. However, the thermomechanical history of the alloys is not available since it remains proprietary and thus further hypotheses or proposal of mechanisms cannot be provided. In addition the full characterization of different phases requires further analysis with TEM/EDX, XRD, and other advanced experimental testing.

The differences that were evident between base and wing parts for In-Ovation L and STb brackets, is a finding that it is in accordance with previous research for two-piece brackets (Eliades *et al.*, 2003, Zinelis *et al.*, 2003, Gioka *et al.*, 2004). In principle the concept is that the surface in contact with the wire should be stiff enough to minimize elastic strain and, simultaneously, hard enough to resist wear by the loads generated by the activated wire. Contrary, the part in contact with enamel should be easily deformed to facilitate the bracket removal after the end of orthodontic treatment (Eliades *et al.*, 2008). The two ferrous brackets followed this concept for both hardness and modulus of elasticity. The results of hardness are very close to previously mentioned values for hardness, with the same alloy used for the production of wing parts (Darabara *et al.*, 2007). However, previous data for the modulus of elasticity are not available in the literature.

Incognito is a one-piece bracket with the same structure in wing and base regions, which is produced by casting (Wiechmann, 2002). Microstructure analysis showed that Incognito consists of three different phases and hence increasing the concerns for possible galvanic action, which also might apply for the base of In-Ovation L. Previous studies have shown that great mismatch in E_{corr} (corrosion

potential) can trigger galvanic action at least under experimental conditions (Siargos *et al.*, 2007). Therefore, it is essential to characterize the three-phase structure of these two brackets, which might be appended to microsegregation phenomena or peritectic transformations during solidification. However, these comments cannot be taken as conclusive for the *in vivo* behaviour of these brackets, which could be determined by *in vivo* studies. The aspect of corrosion resistance in galvanic action between Au-based alloy and Ni–Ti is definitely an interesting proposal for further research.

To the best of our knowledge, this is the first time that the instrumented indentation test (IIT) is applied for orthodontic alloys. This technique is a development of traditional hardness where only a single measurement can be drawn. Contrary, in IIT, force and penetration depth are monitored for the entire time that the indenter is in contact with the sample (Figure 3). Although IIT is more commonly used to measure Young modulus and hardness, additional mechanical properties can be measured such as indentation creep, indentation relaxation, and plastic and elastic parts of the indentation work as thoroughly presented in ISO 14577-1. The great advantage of this technique is that dental devices can be tested as final products, bypassing the need for standard specimens (i.e. dumbbell specimens for tensile testing, rectangular strips for bending, etc.) and thus it is anticipated that this technique will find many applications in mechanical property characterization in dental field.

The clinical implications of this study are twofold. In the first place, it deals with the differences in mechanical properties, whereas the other one pertains to the corrosion resistance in galvanic action of bracket-wire system as a whole. Incognito presents lower modulus, which means lower resistance in elastic deformation under the same stresses compared with ferrous alloys and simultaneously lower fretting and sliding wear resistance. The hardness of Ni–Ti archwires ranges from 300 to 430 HV (Darabara *et al.*, 2007), whereas stainless steel ones can

be up to 600 HV (Hunt *et al.*, 1999). The harder archwires, will leave an imprint in the softer bracket slot and the resultant wear will presumably take out some of the activation of the wire and increase friction (Eliades, 2011).

The results of this study showed that the brackets being tested have significant differences in elemental composition, microstructure, and mechanical properties that could potentially influence their clinical performance. Further evidence from retrieved materials could provide clinically relevant information on their intraoral behaviour.

Conclusions

- Incognito and the base of In-Ovation L are multiphase alloys.
- Significant differences exist in microstructure, elemental composition, and mechanical properties among the brackets tested; these might have a series of clinical implications during mechanotherapy.

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